



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of
SHIGEYASU MORIHIRO et al

5 Serial No. 10/669,860 Group Art Unit: 1713
Filed: September 24, 2003 Examiner: SASTRI, SATYA B.
For: THICKENER FOR WATER-BASED VIBRATION DAMPER

10 DECLARATION UNDER 37 C.F.R. 1.132

Being duly sworn, I, Takahiro MIWA, a citizen of Japan,
residing at 5-1-206, Nishimachi, Takatsuki-shi, Osaka
569-0854 Japan, depose and say:

15 I. I am a chemist of Nippon Shokubai Co., Ltd., an assignee
of record of this application, on the subject matters relating
to this application.

I graduated from Osaka Prefecture University, Faculty
of Engineering, Department of Applied Chemistry in March,
20 1992, and also graduated from graduate school of Osaka
Prefecture University, Polymer Chemistry in March, 1994 and
obtained a master degree.

From April, 1994 up till now, I am an employee of Nippon
Shokubai Co., Ltd., and I have been engaged in the research
25 work of emulsion polymer (especially alkali-soluble
thickener) in 1994 up till now at functional Resins Research
Department and Fine & Specialty Chemicals Research Center.

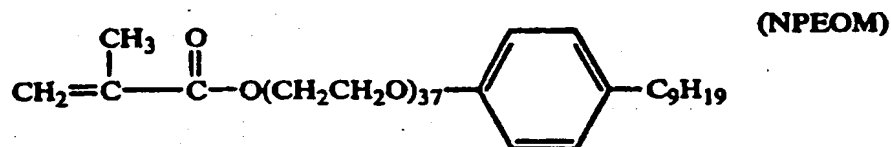
I am well acquainted with all the inventors in this case,
and I am expressing their opinion, as well as my own.

30 II. In order to compare the effects of the above-identified
Morihiro et al application Serial No. 10/669,860 filed on
September 24, 2003 with those of Chang et al patent No.

4,138,381, I made the experiments as follows:

EXPERIMENT I

A polymeric thickener of the composition 86.8%
5 methacrylic acid, 2.6% stearyl methacrylate and 10.6% of a
methacrylate ester of the formula:



was prepared as follows:

10

	Ingredients	Parts by Weight
Charge 1:	Propylene glycol	827
Charge 2:	Methacrylic acid(MAA)	280
	Stearyl methacrylate (SMA)	9
	Propylene glycol	25
Charge 3:	NPEOM	34
	Water	35
	Propylene glycol	111
Charge 4:	Azobisisobutyronitrile	2.2
	Acetone	16
		<hr/> 1339.2

Charge 1 was placed in a 2-liter 3-neck flask equipped
with stirrer, consider, thermometer, nitrogen gas inlet and
outlet and an addition funnel. Charge 1 was blanketed with
15 nitrogen and heated to about 94°C. One-fourth of Charge 2
and one-fourth of Charge 3 and 1/8 of Charge 4 were added
and the temperature held for about 5 minutes. The remainder

of Charges 2 and 3/8 of Charge 4 were mixed and fed simultaneously with the balance of Charge 3 at a constant rate over a period of 80 minutes and then held at 94°C for 15 minutes.

5 The remainder of Charge 4 (1/2) was added in four equal portions at intervals of 30 minutes. After the final addition, the temperature was held 30 minutes, the contents cooled and then discharged.

10 To 300 parts of the above composition was added 150 parts of water and 45 parts of a 30% aqueous ammonia solution. This solution contained about 15% polymeric thickener.

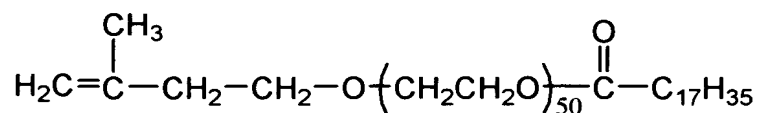
15 The solution thus obtained was colorless transparent uniform solution. Particle size of the solution was measured by dynamic light scattering method using Submicron Particle sizer NICOMP 380 product of NICOMP, but the particles could not be detected.

EXPERIMENT II

20 A four-neck separable flask provided with a stirrer, a thermometer, a refrigerating machine, a nitrogen inlet tube, and a dropping funnel was charged with deionized water (115 parts) and ammonium sulfonate salt of polyoxyethylene dodecyl ether (1.5 parts). The reactants in the flask were kept stirred at an inner temperature of 68°C and swept gently with
25 nitrogen till the interior of the reaction vessel was thoroughly displaced with nitrogen. Then, ammonium sulfonate salt of polyoxyethylene dodecyl ether (1.5 parts) was dissolved in deionized water (92 parts).

30 A mixture of methacrylic acid (40 parts), the combination product of ester with octadecyl polyoxyethylene (3-methyl-3-butenyl) ether (adduct of 50 mols of ethylene oxide) (10 parts), and ethyl acrylate (50 parts) as monomers

for a polymer was introduced into the resultant solution and they were stirred together to prepare a pre-emulsion. Incidentally, the methacrylic acid was the raw material for an alkali-soluble monomer unit, the octadecyl polyoxyethylene (3-methyl-3-butenyl) ether was the raw material for an associating monomer unit, and the ethyl acrylate was the raw material for an ethylenically unsaturated monomer unit. The combination product of ester with octadecyl polyoxyethylene (3-methyl-3-butenyl) ether (adduct of 50 mols of ethylene oxide) had the following structure.



In the reaction vessel, 5% of the pre-emulsion containing the monomers for polymer was stirred for 5 minutes and then sodium hydrogen sulfite (0.017 part) was introduced into the stirred pre-emulsion. Separately, an aqueous solution of polymerization initiator was prepared by mixing ammonium persulfate (0.23 part) with deionized water (23 parts). In the reaction vessel mentioned above, 5% of the aqueous polymerization initiator solution was added and stirred for 20 minutes to effect initial polymerization. The remainder of the pre-emulsion and the aqueous initiator solution were uniformly added dropwise over a period of two hours into the reaction vessel while the inner temperature of the reaction vessel was kept at 72°C. After the dropwise addition was completed, the dropping funnel was washed with deionized water (8 parts) and the washings were introduced into the reaction vessel. The reaction mass in the reaction vessel was continuously stirred for one more hour with the inner

temperature of the reaction vessel kept at 72°C and then cooled to complete the reaction. Consequently a thickener for vibration damper 1 having a non-volatile content of 30% was obtained.

5 The thickener was milky white emulsion. The emulsion was measured by dynamic light scattering method using Submicron Particle Sizer NICOM 380 product of NICOMP to find 80nm.

10 III. CONCLUSION

As being clear from the above-identified Experiment I, the produced solution as the thickener was colorless transparent uniform solution and particles was not found by dynamic light scattering method, so it was not emulsion.

15 About other product (solution) as thickener except Example 2 in Chang et al, monomer compositions, solution compositions and polymerization procedures are almost of the same as Example 2, so it can be judged that the other products are not emulsion.

20 On the contrary, the thickener used in Morihiro et al application Serial No. 10/669,860 is emulsion as being clear from Experiment II.

Therefore, both are quite different in the thickeners each other.

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The undersigned Takahiro MIWA declared that all the statements made herein are true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or
30 imprisonment, or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardized the validity of the application or any patent

issuing thereon.

Dated this 21st day of August, 2006.

5

By Takahiro Miwa
Takahiro MIWA